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<p>The research carried out in this program has focused on the analyses of the transport, kinetic, and thermodynamic processes taking place during diamond deposition in direct current and hot-filament reactors. The research carried out may be grouped into four broad categories: (1) development of multistep chemical mechanisms for the deposition of diamond, graphite, and point defects; (2) development and execution of detailed reactor-scale models; (3) development and execution of simplified, analytical reactor-scale models; and (4) development of kinetic and thermodynamic models for diamond nucleation and the early stages of growth.</p>			
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**Materials Processing and Manufacturing Technologies
for Diamond Substrate Multichip Modules (DSMCM)***

Final Report
Contract N00014-93-2002
February 26, 1997

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I. Summary

The research carried out in this program has focused on the analyses of the transport, kinetic, and thermodynamic processes taking place during diamond deposition in direct current arcjet and hot-filament reactors. The research carried out at Colorado State University has been theoretical in nature, and has been executed in close collaboration with research staff at The Norton Company, Sandia National Laboratories, the Naval Research Laboratory, and Texas Instruments.

The research carried out by the PI and his students may be grouped into four broad categories:

1. Development of multistep chemical mechanisms for the deposition of diamond, graphite-like materials, and point defects;
2. Development and execution of detailed reactor-scale models;
3. Development and execution of simplified, analytical reactor-scale models;
4. Development of kinetic and thermodynamic models for diamond nucleation and the early stages of growth.

In the following sections, principal accomplishments from this program will be described, followed by a list of publications resulting from the research.

II. Surface Chemistry

1. deposition mechanism

When this contract began, several deposition mechanisms had been proposed by different investigators. These mechanisms utilized a single gas-phase precursor species, CH_3 or C_2H_2 , and accounted for growth of a single phase, diamond. Recognizing that reactor systems can produce different concentrations of potential precursor species depending on the operating conditions, we assembled a growth mechanism that allowed for simultaneous growth of diamond and a graphite-like phase from C_2H_2 , CH_3 , and C. The mechanism, while detailed, is not elementary to the extent of describing the specific bond breaking and formation on a diamond (100) surface. In fact, the unreconstructed (100) dihydride surface almost certainly does not exist. This will be discussed in §II.2 below. Instead, the mechanism includes systematic steps for the deposition of the three gas-phase hydrocarbon species. The mechanism consists of 25 surface reactions which may be broken down into different categories: initiation, recombination, methyl addition, acetylene addition, carbon atom addition, and graphite reactions.

None of the required rate constants for the surface reactions are directly available from experiment. In many cases, rate constants from other models of diamond growth have been used, and in others, we assign the same rate constant to each reaction of analogous type. All reactions in the mechanism are reversible, with the rates for the reverse reactions obtained from thermochemistry.

The first step in the surface mechanism is the abstraction of a surface H atom by a gas-phase H atom. After the abstraction, a reactive radical site is left on the surface, as illustrated in Figure 1. This initiation step is common to most kinetic schemes for diamond growth proposed in the literature. The surface radical sites can be terminated efficiently via reactions with gas-phase H atoms. Termination reactions should be much more facile than the initiation reaction. Methyl radicals, carbon atoms, and acetylene molecules from the gas can also add to the surface radical, although with a lower probability than an H atom. An adsorbed CH_3 group is shown in the figure. Subsequent reactions involve the formation of bonds between the added hydrocarbon group and neighboring surface carbons. When an added carbon has formed four lattice bonds with neighboring carbon atoms it is said to be fully incorporated and is then part of the diamond lattice.

The surface mechanism is coupled to reactor scale models and used to predict growth rates of diamond and graphite. These calculations are discussed in §III.

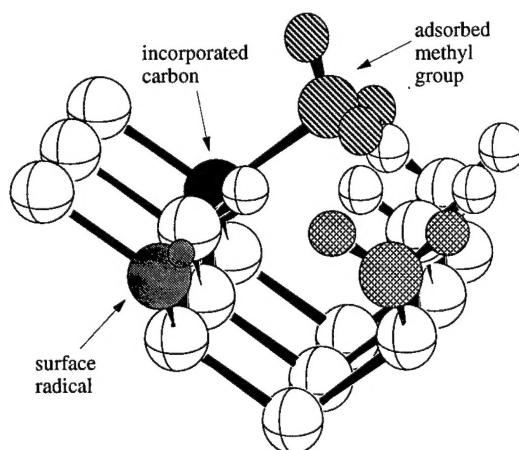


Figure 1. Identification of surface species involved in (100) deposition mechanism.

2. dimer growth mechanism

Data from AFM and STM strongly indicate that surfaces such as (100) do not remain unreconstructed, even under growth conditions where large atomic hydrogen fluxes exist. Consequently, a detailed surface kinetics mechanism has been developed to describe the growth of diamond on a (100)-(2×1) surface. The mechanism is initiated by the abstraction of a hydrogen, and growth occurs through the adsorption of a carbon species, such as CH₃. A typical model structure for this mechanism, used to compute the thermophysical properties, is shown in Figure 2. The thermodynamic properties of all of the structures appearing in the proposed mechanism have been computed using the molecular mechanics program MM3(92). The temperature-dependent quantities predicted by MM3—enthalpy, entropy, and heat capacity—have been fit to polynomials over the temperature range 298.15 $\leq T \leq$ 2000 K. These data have been used to study the kinetics of the growth process.

Carbon-bearing molecules can add to the (100)-(2×1) surface by bridging across the troughs formed by two adjacent dimer bonds, or inserting directly into a dimer bond. The model is constructed in such a way that the dimer structure repeats itself as the surface “grows,” with the dimer pairs oriented 90° relative to the previous layer. There are 44 unique structures (surface species) present in the model, with over 100 reactions describing abstraction, termination, insertion, adsorption, and isomerization. The model has been tested for self-consistency and is included in reactor scale models of diamond growth.

3. reduced deposition mechanism

The detailed deposition mechanisms described in §§II.1 and II.2 have been incorporated into one- and two-dimensional reactor models which predict the flow, temperature, and composition fields within a diamond reactor. The full reactor models are very detailed and may require

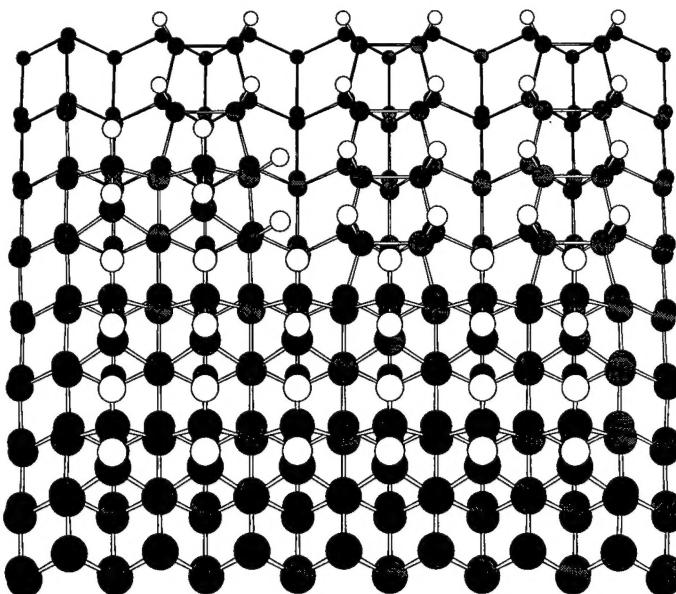


Figure 2. Model dimer structure used to compute thermophysical properties of diamond.

significant computational expense to execute. Part of an effort to develop simplified reactor models has been to reduce the complexity of the deposition mechanism.

Much theoretical analysis has been done on the elementary heterogeneous chemical reactions leading to diamond deposition. There will likely never be full agreement on the details of the process. However, it has been found that relatively few reactions are needed if the goal is limited to an accurate description of deposition rates. Earlier modeling work showed that CH_3 , C, or a combination of the two could be responsible for bringing carbon to the surface, depending on reactor type and operating conditions. It has also been observed that, depending on growth conditions, point defects are incorporated into the lattice.

By examining the detailed mechanisms developed in this project and those proposed by others, it has been found that accurate estimates of growth rate can be obtained when the deposition mechanism is reduced to a sequence of 9 irreversible reactions with 4 surface species and 2 bulk phases, diamond and “defect”. Further, it is possible to analytically solve the rate expressions for this set of reactions to obtain closed form expressions for diamond growth rate and point defect formation rate. The resulting expressions are included in simplified reactor models discussed in §IV.

III. Reactor Modeling

Two basic reactor types have been modeled in this project: direct current (dc) arcjet and hot-filament. Although the physics of momentum, energy and mass transport are very different between the two reactor types, to leading order both systems may be approximated as idealized stagnation flows. Using this approximation it is possible to reduce the three-dimensional governing conservation equations to a set of one-dimensional equations, and it is therefore possible to include arbitrarily complex gas-phase and surface chemistry mechanisms into the models. The overall goal of the reactor modeling is to investigate the effect of reactor operating conditions on diamond growth rate and graphite and defect formation rate. Also, through investigation of parameter space it is possible to identify dominant transport and kinetic mechanisms and use this insight to develop simplified reactor models.

1. dc arcjet reactors

One reactor type in which investigators have been able to achieve high growth rates over relatively large areas is the dc arcjet system. In this reactor type, high growth rates result from very large fluxes of material— H_2 and often Ar—passing through a plasma torch and jetting toward the deposition surface. Computations have been performed to study the effects of inlet H concentration, inlet CH_4 concentration, inlet temperature, and CH_4 injector location on diamond growth rate and film quality.

The degree of H_2 dissociation within the plasma torch has a direct consequence on both growth rate and film quality. The distribution of gas phase hydrocarbon species depends strongly on the mole fraction of H at the plasma torch exit. Even when little H is formed in the torch, that which is available quickly abstracts a hydrogen atom from CH_4 to form CH_3 , until sufficient CH_3 is formed to bring CH_4 and CH_3 into partial equilibrium. As more H is formed in the plasma torch, more hydrogen is abstracted from the injected CH_4 , until an appreciable amount of atomic carbon is formed. Thus, as illustrated in Figure 3, there is a correlation between the precursor species that dominates growth and the amount of H formed in the plasma torch. The figure also demonstrates that the predicted diamond growth rate rises monotonically with H concentration. The results shown in Figure 3 correspond to an inlet CH_4 concentration of 0.5%, a plasma torch exit temperature of 2500 K, and a substrate temperature of 1200 K. The contributions of C and CH_3 as growth precursors directly tracks the relative amounts of these species formed in the gas phase.

When the amount of inlet CH_4 is varied, the predicted growth rates for diamond and graphite-like phases follow the expected trend: diamond achieves a maximum growth rate at approximately 1% CH_4 , while the growth rate of the graphite-like phase monotonically increases. The reason for the turnover in diamond growth rate is the increased conversion of CH_4 to C_2H_2 in the gas phase. Although the acetylene does react with the surface, it is predicted to do so at an appreciably lower

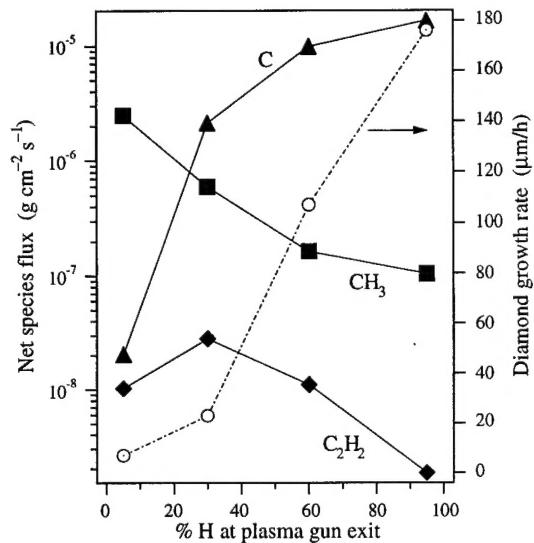


Figure 3. The net flux of each growth precursor and the total growth rate as a function of the inlet H concentration.

rate than CH_3 , and the decrease in CH_3 through conversion to C_2H_2 results in a lower overall growth rate.

A means of improving carbon utilization in dc arcjet systems is to exploit the disparate time scales for the physical processing occurring within the reactor. Characteristic time scales associated with convection of mass, energy, and momentum, as well as diffusion of these quantities (transport time scales) compete with the characteristic time scales associated with each of the homogeneous and heterogeneous reactions taking place in the reactor (chemical kinetic time scales). It may be possible to exploit the competition between the different scales to increase diamond growth rate for a given set of operating conditions. A means of implementing this concept is to control the location of the hydrocarbon injector. Experiments and calculations have been performed to investigate two relative extremes for hydrocarbon injection. For the first, premixed injection, the methane is introduced outside of the plasma jet, near the exit of the plasma torch; a combination of entrainment and diffusion transports the carbonaceous species into the stagnation flow at the substrate. The hydrocarbon spends more time at lower temperatures as it is transported to the substrate, temperatures at which C_1 species are thermodynamically favored over C_2 species. In the second injection strategy, methane is introduced inside the boundary layer, that is, within several millimeters of the substrate. The relatively short residence time within the boundary layer and the lower gas temperatures near the substrate inhibit the conversion of methane to acetylene through methyl and methylene radical routes, ideally having time only to produce CH_3 . A series of boundary-layer calculations were made to investigate the substrate injection strategy. These numerical results, together with the experimentally-obtained growth rates, are shown in Figure 4. It was found that, for substrate injection, higher CH_4 concentrations could be used than for other premixed injection strategies, and the quality of the film remained at an acceptable level.

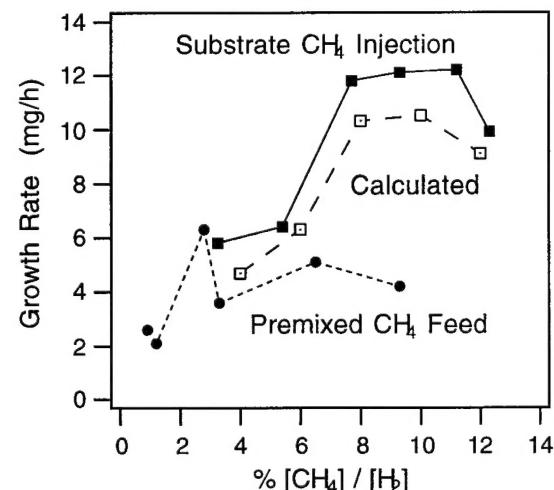


Figure 4. Measured and calculated mass deposition rates for two CH_4 feed gas methods. Calculated rates correspond to substrate injection.

2. *hot-filament reactors*

In hot-filament reactors diamond is typically grown by slowly flowing a reactant mixture—usually H₂ and CH₄, sometimes with small amounts of O₂—past one or more glowing tungsten wires towards a temperature-controlled substrate. Power is supplied to the filament to maintain a temperature in the range 2000–2800 K, while the substrate is either actively or passively kept between 900 and 1200 K. Analysis of diamond growth in hot-filament systems is complicated by several factors. First, H₂ dissociation occurs via heterogeneous reactions on the filament. The specifics of these reactions are not known. Second, for the low pressures used in most hot-filament reactors, there is an apparent temperature discontinuity between the filament and the adjacent gas that arises from noncontinuum effects. Finally, the injected hydrocarbon may adsorb onto the filament, acting to “poison” the surface and reducing the effectiveness of the filament for dissociating hydrogen.

All of these complicating factors have been taken into account in a one-dimensional stagnation model of a hot-filament reactor. The model results are compared directly against quantitative measurements of near-substrate gas composition obtained by molecular beam mass spectroscopy. Using a simple model of filament poisoning it has been found that good agreement exists between prediction and measurement for all major gas-phase species. The model also predicts the measured film growth rate to within 50%.

IV. Simplified Models

Through continued use of the detailed models described in §III for the simulation of actual dc arcjet reactors, and detailed analysis of transport and chemical kinetic processes taking place during diamond growth in these highly convective environments, it has been demonstrated that the dominant physics of the gas phase and gas-surface processes can be captured by making specific and restrictive assumptions regarding the behavior of the system. The motivation of this work is development of a simplified model of the processes occurring in a dc arcjet reactor, a model free of numerical instabilities or convergence problems, and one that runs in near real-time. The simplified model developed in this project executes in less than a millisecond on a fast workstation, approximately 10⁵–10⁶ times faster than the detailed numerical model discussed in §III. Given a specific set of reactor operating conditions, the model predicts heat flux to the substrate, diamond growth rate, and point defect fraction in the diamond phase.

The natural division of the physical system into four characteristic domains—plasma torch, free stream, boundary layer, and surface—led to the development of simplified thermodynamic, transport, and chemical kinetic models for each of the four regions. The plasma torch is modeled using a very simple thermodynamic model, which is based upon the treatment of the plasma torch as a well-mixed, steady, open-flow system. The actual energy input to the system through the arc discharge is modeled as a heat flux into the system. The plasma torch model is constructed such that the outlet temperature of the gas mixture is specified, as is the power input to the torch. The thermodynamic model then predicts the necessary molar flow rate of H_2 input to the plasma torch, and the degree of dissociation of H_2 occurring in the torch. The plasma torch model may also be formulated such that it is assumed that H and H_2 reach chemical equilibrium at the torch exit conditions for fixed inlet H_2 flow, and torch power is then computed so that the first law is satisfied. As an example of this, a reactor pressure of 30 Torr is assumed and the H mole fraction (due to H_2 dissociation) and plasma torch power are computed. As shown in Figure 5, both of these quantities increase dramatically as the gas mixture temperature increases. By assuming that an increased exit temperature is achieved *and* that the mixture reaches chemical equilibrium, and great demand for energy (torch power) is required.

The free stream region, the portion of the system between the plasma torch exit and the top of the boundary layer, is characterized by a high-velocity, moderately high Reynolds number flow. As a consequence of the known hydrocarbon combustion kinetics in the diamond system, the gas-phase hydrocarbon species are assumed to exist in a pseudo-chemical equilibrium state until the gas reaches the boundary layer. Within the boundary layer, H undergoes heterogeneous reactions which efficiently remove it from the gas phase. The sharp drop in H concentration between the free stream and the substrate strongly affects the hydrocarbon composition within the boundary layer. The simplified deposition mechanism described in §II.3 is used to predict the diamond growth rate and point defect fraction.

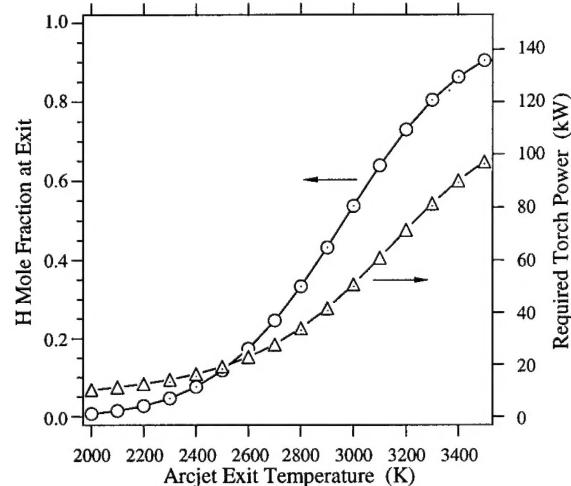


Figure 5. The fraction of the gas leaving the torch as atomic hydrogen and the torch power required to achieve this degree of dissociation, for fixed inlet H_2 flow rate, as a function of desired gas temperature.

V. Nucleation and Morphology

Experimental observations have demonstrated that the formation of an intermediate layer [diamond-like carbon (DLC), amorphous carbon (a-C), carbides, or graphite] on nondiamond substrates prior to diamond nucleation is a requisite step in most spontaneous nucleation processes. The existence of the incubation period frequently observed in diamond nucleation may be related to interactions of gas-phase species with the substrate surface. The events occurring during the incubation period and the initial nucleation stage may include (i) impingement, (ii) accommodation, (iii) adsorption and desorption, (iv) surface diffusion, and (v) diffusion to/from the substrate bulk. The rearrangement of atoms in steps (iv) and (v) may lead to the formation of an intermediate layer on the substrate, whereas carbon clusters embedded in or located on the interlayer may form diamond nuclei depending on the energy, rate, and size conditions.

A model has been developed to examine theoretical aspects of diamond nucleation on carbide-forming substrates in an attempt to address the incubation period and the transient nucleation stage during CVD. The starting point of the model is atomistic theory because of the nature of the nonclassical nucleation in diamond CVD. The Volmer-Weber 3-D island nucleation and growth mode is considered to be the nucleation mode of diamond on nondiamond substrates due to diamond's extraordinarily high surface energy. Incorporating the physical phenomena in steps (i)–(v) above, it is possible to write kinetic expressions for the surface concentrations of single atoms and clusters. Using deposition conditions consistent with dc arcjet systems such as those modeled in §§III and IV, the time dependent surface concentration of single atoms has been computed for Si, Ta, W, Mo, Fe, and Ti substrates. Figure 6 illustrates the calculated time evolution of the surface concentration of single C atoms on the different substrates using an assumed mean residence time of 10^{-6} s. Exploratory calculations indicate that the mean residence time may range from less than 1 ns to hours, depending on the adsorption state, adsorption energy, and substrate temperature; the residence time used here is a median value of times considered to be reasonable. The results in Figure 6 show that, the larger the larger the diffusivity of C atoms into the substrate, the smaller the surface concentration of C atoms. On W, Ta, and Si substrates, the surface C concentration attains its saturation value quickly (< 100 ms) after exposure to a C atom flux. Although the diffusivity of C in Fe is higher than in other substrate materials, the incubation period on Fe is short. This is due to the relatively high surface mobility of C on Fe, and the result

suggests that the diffusion of C atoms into a substrate is not a critical factor governing the incubation period.

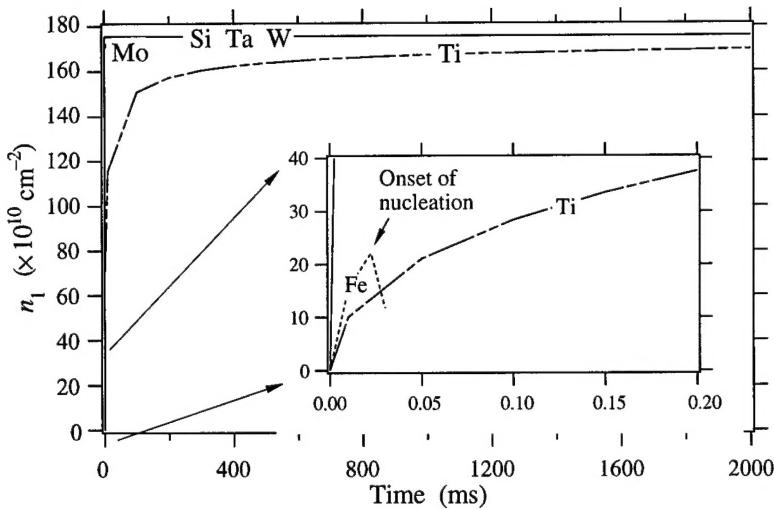


Figure 6. Time evolution of the surface concentration of single C atoms on a Si, Ta, W, Mo, Fe, and Ti substrate.

Silicon is one of the most common substrate materials for diamond deposition. Heteroepitaxial nucleation of diamond on silicon wafers may take place through the formation of an intermediate β -SiC epitaxial conversion layer on the silicon surface due to crystallographic registry across the interface. Epitaxial nucleation of diamond on Si(100) wafers has been demonstrated to occur via the formation of an epitaxial β -SiC conversion layer that formed during the in situ carburization step. The β -SiC(111) layer is known to form as an intermediate layer on a Si(111) substrate due to the epitaxial relationship between silicon and β -SiC. In this project, and mechanism for the nucleation of diamond-phase carbon clusters on β -SiC(111) is examined by analyzing energetically and entropically favorable pathways for hydrocarbon deposition. The β -SiC surface is assumed to be completely hydrogenated and therefore in an unreconstructed state. The critical size and structure of diamond phase carbon clusters formed on β -SiC has been computed based on the nucleation mechanism. We have found that feasible routes for the formation of propane-like clusters may be achieved from CH_3 gas phase precursors. However, it was not possible to find a combined $\text{C}_2\text{H}_2/\text{CH}_3$ deposition mechanism that was entropically favorable. Using the mechanisms for the formation of the propane-like kernels, it was found that critical cluster sizes ranged from 7 to 20 carbon atoms.

Another area of investigation in this project is the chemistry of the interlayer formed during diamond deposition. A thermodynamic quasiequilibrium (QE) model has been used to examine

the importance of various hydrocarbon species to interlayer formation, and also to quantify the effect of varying thermodynamic parameters, such as temperature, pressure, and CH_4/H_2 ratio on the growth rate of the solid phase (the substrate carbide, graphite, or diamond). Through application of the QE model to the substrate carbide, graphite, and diamond system, the desorption rates of volatile species and the deposition rates of the substrate carbides/solid carbons are calculated for varying substrate temperature, reactor pressure, and inlet CH_4 mole fraction. Using these results, a phase diagram may be obtained for the substrate carbides, graphite, and diamond.

The QE model predicts that, for Ti, Ta, Si, Mo, Fe, and W substrates, the most stable form of carbide/carbon to form immediately on the substrate surface would be TiC , TaC , SiC , Mo_2C , Fe_3C , and WC , respectively. Diamond and graphite are not likely to deposit on the substrate for the growth conditions—substrate temperature, pressure, and inlet CH_4/H_2 ratio—corresponding to the region lying to the left of the graphite phase/stability curve shown in Figure 7. This is because the graphite and diamond stability regions lie within the TiC , TaC , SiC , Mo_2C , Fe_3C and WC stability regions. If the process conditions during diamond CVD correspond to the region lying between the diamond and graphite stability curves, then the various carbides as well as graphite are equally likely to form since the diamond stability curve lies within the stability regions for graphite and the carbides. If the processing parameters are such that the conditions correspond to a region lying to the right of the diamond stability curve, then the formation of diamond, graphite, and the carbides are equally probable from a thermodynamic standpoint.

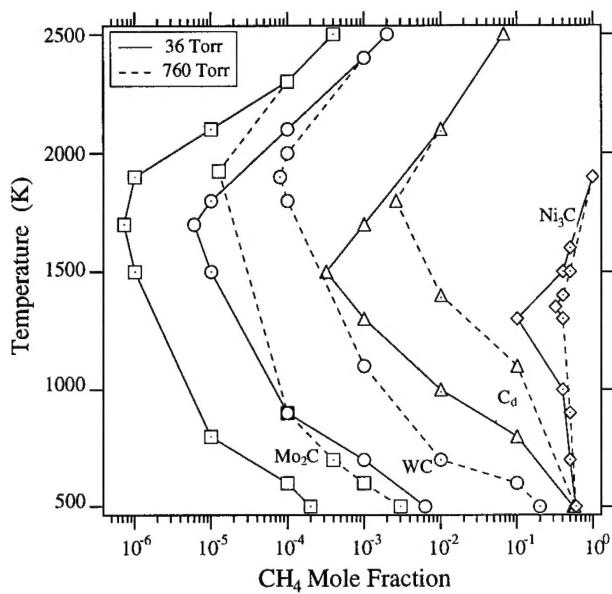


Figure 7. Calculated phase diagrams for Mo_2C , WC , diamond (C_d), and Ni_3C at 36 Torr and 760 Torr.

VI. Publications

This project resulted in 16 articles in refereed journals, 28 presentations, 1 book, and 1 book chapter. Listed below are the journal and book citations.

Journal Articles

1. "Analysis of diamond growth in subatmospheric dc plasma-gun reactors," M.E. Coltrin and D.S. Dandy, *J. Appl. Phys.* **74**, 5803 (1993).
2. "Computational simulation of diamond chemical vapor deposition in premixed $\text{C}_2\text{H}_2/\text{O}_2/\text{H}_2$ and CH_4/O_2 strained flames," E. Meeks, R.J. Kee, D.S. Dandy, and M.E. Coltrin, *Combustion and Flame* **92**, 144 (1993).
3. "Diamond growth using remote methane injection in a direct current arcjet chemical vapor deposition reactor," S.W. Reeve, W.A. Weimer, and D.S. Dandy, *Appl. Phys. Lett.* **63**, 2487 (1993).
4. "Effects of temperature and filament poisoning on diamond growth in hot-filament reactors," D.S. Dandy and M.E. Coltrin, *J. Appl. Phys.* **76**, 3102 (1994).
5. "Relationship between diamond growth rate and hydrocarbon injector location in direct-current arcjet reactors," D.S. Dandy and M.E. Coltrin, *Appl. Phys. Lett.* **66**, 391 (1994).
6. "Experimental measurements and numerical simulations of the gas composition in a hot-filament-assisted diamond chemical-vapor-deposition reactor," M.C. McMaster, W.L. Hsu, D.S. Dandy, and M.E. Coltrin, *J. Appl. Phys.* **76**, 7567 (1994).
7. "Dependence of the gas composition in a microwave plasma-assisted diamond chemical vapor deposition reactor on the inlet carbon source: CH_4 and C_2H_2 ," M.C. McMaster, W.L. Hsu, D.S. Dandy, and M.E. Coltrin, *Diam. Related Mater.* **4**, 1000 (1995).
8. "Molecular beam mass spectrometry studies of chemical vapor deposition of diamond," W.L. Hsu, M.C. McMaster, M.E. Coltrin, and D.S. Dandy, *Jpn. J. Appl. Phys.* **33**, 2231 (1994).
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10. "A simplified analytical model of diamond growth in direct current arcjet reactors," D.S. Dandy and M.E. Coltrin, *J. Mater. Res.* **10**, 1993 (1995).
11. "On the optimization of a dc arcjet diamond CVD reactor," S.W. Reeve, W.A. Weimer, and D.S. Dandy, *J. Mater. Res.* **11**, 694 (1996).
12. "Nucleation kinetics of diamond on carbide-forming substrates during CVD," H. Liu and D.S. Dandy, *J. Electrochem. Soc.* **143**, 1104 (1996).
13. "Momentum and thermal boundary-layer thickness in stagnation flow chemical vapor deposition reactors," D.S. Dandy and J. Yun, *J. Mater. Res.* **12**, 325 (1996).
14. "A quasi-equilibrium model for the prediction of interlayer chemistry during diamond chemical vapor deposition," P. Mahalingam and D.S. Dandy, *Thin Solid Films*, in press, 1996.

15. "A model for the nucleation of diamond clusters on Si(111) substrates," P. Mahalingam, H. Liu, and D.S. Dandy, *J. Appl. Phys.* **81**, 1966 (1997).
16. "Simulation of morphological instabilities during diamond chemical vapor deposition," P. Mahalingam and D.S. Dandy, *Diam. Related Mater.*, in press, 1996.

Books and Book Chapters

1. *Diamond Chemical Vapor Deposition Processes: Nucleation and Early Growth Stages*, H. Liu and D.S. Dandy, Noyes Publications, Park Ridge, New Jersey (1995) .
2. *Diamond Thin Films Handbook*, Chapter 4, "Deposition chemistry: deposition pathways, nucleation, and growth, D.S. Dandy and M.E. Coltrin (D.K. Reinhard and J. Asmussen, eds.), Marcel Dekker, Inc., New York, in press (1996).